

**R E M A R K S**

The amendments made to the specification by this current response do not add new matter to the specification. In particular, applicants note that the added paragraphs are derived from U.S. Serial No. 09/088,300 (now issued U.S. Patent No. 6,068,861). See U.S. Patent No. 6,068,861, column 3, lines 20-26, and column 4, line 32 – column 6, line 22. U.S. Serial No. 09/088,300 and U.S. Patent No. 6,068,861 were incorporated by reference in their entirety by the current application as originally filed. See page 1, lines 4-6, page, 2, lines 15-25, and page 20, lines 19-20 of the current application; see also, page 1 of the Preliminary Amendment filed with the original application (amending the specification to indicate that U.S. Ser. No. 09/088,300 is “now issued as United States Patent No. 6,068,861).

Claims 1-10 are currently pending in the present application. Claims 1-5 were indicated as allowable by the Examiner since the “prior art does not teach or suggest producing a concentrated stabilized biocidal composition which comprises adding BrCl to an overbased, aqueous alkali metal sulfamate solution, wherein the pH of the aqueous alkali metal sulfamate solution results in the biocidal composition having a pH of at least 7.” See Office Action, dated 5/21/07, pages 10-11. Nevertheless, by the current amendment, claims 1-5 are cancelled (without prejudice to pursue this subject matter in a later application) in favor of newly added process claims 11-20 and product claims 21-30. Applicants submit that process claims 11-20 are narrower than cancelled claims 1-5, and therefore also free of the prior art.

**Regarding Support for Newly Added Claims**

Support for newly added claims 11-30 can be found in the general teachings and specific examples of the current specification (including relevant portions of U.S. Patent No. 6,068,861 added to the present application by the current amendment). Claims 11, 16, 21 and 26 find support specifically in original claims 1 and 4, and at p. 9, line 17 – page 10, line 3 of the present application. Claims 12-15, 17-20, 22-25 and 27-30 find support in claim 5 and at p. 10, lines 4-9. Specifically, at page 10, lines 4-9, the current application refers to feeding two streams (one containing bromine chloride, the other an alkali metal sulfamate solution such that “the atom ratio of nitrogen to active bromine from these feed streams is greater than 0.93, and more

preferably in the range of about 1.0 to about 1.4.” Moreover, a footnote to Table 2 of U.S. Patent 6,068,861 (see column 4, line 60), which was incorporated by reference refers to this ratio as the “sulfamic acid to halogen mole ratio.” It is clear from a review of the Examples that when the “halogen” is bromine chloride, “halogen” refers to the sum of chlorine and bromine used to make the bromine chloride. Thus, it is noted that the sulfamic acid to halogen ratio used in Example 3 of the ‘861 patent (column 5, lines 5-16) was 0.93 (calculated from weights listed in Example 3, which is a slight deviation from the value of 0.92 indicated in Table 2) and in Example 7 of the ‘861 patent (column 6, lines 12-22) this ratio was 1.41.

#### Regarding Patentability Over the Prior Art

As noted above, claims 1-5 were allowed by the Examiner. Applicant submits that claims 11-20 are all narrower than original claim 1 and thus also free of the prior art.

Claims 6-10 were rejected under 35 U.S.C. § 102(b) as being anticipated by Goodenough (USPN 3558503). See page 2 of the Office Action dated 5/21/07. Claims 6-10 were also rejected under 35 U.S.C. § 102 over Goodenough (USPN 3558503), and under 35 U.S.C. § 103 over Goodenough in view of expert declaration testimony submitted during the interference in which the current application was involved. See Office Action, dated 5/21/07, page 6. Specifically, the Examiner (following the recommendation made by the Board) has taken the position that the language in claim 6 reciting a “stabilized, bromine-based biocide,” must be construed in view of the specification “to mean the bromine compound resulting from the recited steps, as opposed to the resulting aqueous formulation or solution containing the bromine compound.” See Office Action, dated 5/21/07, sentence spanning pages 2-3, and page 7, lines 3-8. Stated succinctly, the Examiner’s position is that claims 6-9 are directed solely to a stabilized bromine-based compound (i.e., N-bromosulfamate) or use thereof (in the case of claim 10), rather than an aqueous formulation, solution or composition containing the bromine compound. Regarding Applicants’ arguments (made during the interference) as to nonobvious differences between the subject matter of claim 6 and Goodenough, the Examiner (again repeating statements made by the Board) states:

Instant invention alleges that the use of bromine chloride in lieu of bromine results in a nonobvious difference. This contention lacks merit for a

number of reasons. First, instant independent claim 6 does not recite any pH values. Second, instant claims do not call for a composition of compounds or mixture of N-halo compounds but instead are directed to any "stabilized, bromine-based biocide" that is formed by the recited process steps. Third, nowhere does the instant specification point to any experimental evidence establishing any difference, let alone a patentable difference, between a biocide within the scope of instant claim 6 and Goodenough's final bromine compound where sodium chloride or calcium chloride is used as a starting material along with bromine. Note it is inherent that the combination of bromine with sodium chloride or calcium chloride in the Goodenough patent would result in the BrCl compound employed in instant invention.

See Office Action, dated 5/21/07, paragraph spanning pages 4-5. Taking into account that Applicants' arguments made during the interference were premised upon a reading of claim 6 as being directed to a composition, as opposed to a compound, Applicants respond to the Examiner's contentions in turn.

Regarding the first contention, Applicants note that newly added claims 21-30 each recite a minimum pH of "at least about 13.0" for an alkali metal sulfamate solution during addition of bromine chloride.

Regarding the second contention, Applicants specifically note that the subject matter of newly added claims 21-30 is an aqueous biocidal solution rather than a stabilized bromine-based biocidal compound. That is, with these claims Applicants seek a scope of patent protection which is not merely limited to a stabilized halogen compound, or limited to an aqueous biocidal solution produced by the process recited in these claims. Rather, Applicants seek a scope of patent protection encompassing any aqueous biocidal solution exhibiting substantially equivalent mixtures of stabilized halogen species and additionally in the case of claims 23, 25, 28 and 30, concentrations of active stabilized halogen.

Regarding the third contention, Applicants recognize that patentability over the prior art depends on whether the aqueous biocidal solutions produced by the methods recited in these claims are novel and nonobvious over the prior art. Accordingly, to assist the Examiner in this assessment, Applicants direct the Examiner's attention to ¶¶ 1-12 of Appendix A, which is titled Second Declaration of B. Gary McKinnie. This declaration was submitted during the interference in which the current application was previously involved. In ¶¶ 4-12, Dr. McKinnie discusses the composition of an aqueous biocidal solution produced in accordance with the

methods recited in claims 21-30. In laboratory experiments conducted during his employment with Albemarle (described in ¶¶ 4-10), Dr. McKinnie determined that addition of bromine chloride to an alkali metal sulfamate solution which was maintained at a pH of at least 13.0 during the addition of the bromine chloride results in an aqueous biocidal composition comprising an about 4:1 molar ratio of stabilized bromine (i.e., N-bromosulfamate) to stabilized chlorine (i.e., N-chlorosulfamate). See in particular, ¶ 8.

Goodenough describes methods for preparing N-bromosulfamate solutions. According to Goodenough, one method for preparing these solutions is to add bromine to an alkaline metal sulfamate solution. Goodenough further teaches that N-bromosulfamate solutions can be prepared even using as a starting material a brine solution containing sodium chloride or calcium chloride; however, there is no indication in Goodenough that N-chlorosulfamate would be produced. Applicants note that in order for N-chlorosulfamate to form in an alkali solution, chloride would first have to be oxidized to hypochlorite by bromine or hypobromite. Applicants are not aware of any scientific reference which indicates that this reaction can occur, and request that if such a reference exists, that the Examiner provide such a source. Indeed, as noted at p.1, lines 13-16 of the present application, prior art methods for producing hypobromite rely on the reverse reaction (i.e., on the oxidation of bromide to hypobromite by hypochlorite). See also, ¶ 11 of Dr. McKinnie's Declaration submitted during the interference (attached as Appendix A to the present Response). Applicants agree that the stabilized bromine compound in Goodenough's composition, and Applicants' aqueous biocidal compositions is the same (i.e., N-bromosulfamate). Nevertheless, the aqueous biocidal compositions of newly added claims 21-30 also comprise N-chlorosulfamate and thus differ from the compositions produced by Goodenough, which only comprise N-bromosulfamate.

In view of the above arguments, Applicants submit that claims 11-30 are patentable over the prior art. Consideration and allowance are respectfully solicited.

In the event the Examiner believes an interview might serve in any way to advance the prosecution of this application, the undersigned is available at the telephone number noted below.

Application No.: 09/785,890

Docket No.: SU-7073-L

The Office is authorized to charge any necessary fees to a deposit account.

Dated: November 20, 2007

Respectfully submitted,  
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**SECOND DECLARATION OF B. GARY McKINNIE**

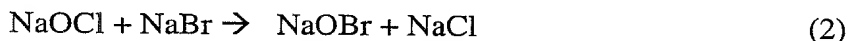
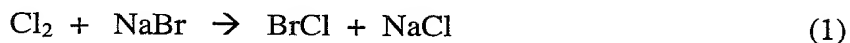
I, B. Gary McKinnie, declare as follows:

1. I am the same B. Gary McKinnie who provided a previous declaration for interference nos. 105,222, 105,223 and 105,230.
2. I have held various technical titles within Ethyl / Albemarle Corp. (Chemist, Senior chemist, Specialist, Senior Specialist, Advisor, Senior Advisor) and in 2000 was promoted to Distinguished Advisor.
3. Since 1983 I have conducted research and development of processes for producing bromine, brominated flame retardants and other bromine-related products. Since 2000 I have carried out research and development work on processes for making bromine-based biocides.
4. In 2000 as part of my research duties at Albemarle I personally conducted lab experiments to determine the N-halo compound make-up (specifically, the presence of N-bromosulfamate and N-chlorosulfamate) of a biocide solution which results from adding bromine chloride (pre-prepared or made inline) to an aqueous alkali metal sulfamate solution formed from water, sulfamic acid and sodium hydroxide (an alkali metal base). I also experimented adding bromine first, then chlorine, to the same aqueous alkali metal sulfamate solution.
5. The method used to determine the presence of N-halo compounds in the resulting composition was as follows.
  - a. Hach Company (Loveland, CO) has two color tests developed to measure “free chlorine” and “total chlorine” in water at the few part per million (ppm) level. It is based on the chlorine oxidation of N,N-diethyl-p-

phenylenediamines (DPD) to a Wurster dye that is highly colored, the intensity of which is proportional to the amount of oxidation that has occurred. **EXHIBIT 1074**, p. 2, col. 2, ¶¶ 1-2.

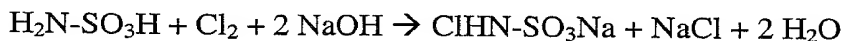
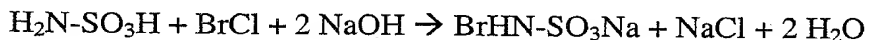
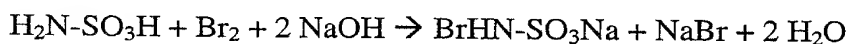
- b. N-chloro compounds are very slow to oxidize DPD but in the presence of a trace of iodide the oxidation does occur rapidly. **EXHIBIT 1074**, p. 3, col. 1, ¶ 1.
  - c. While N-chloro compounds are very slow to oxidize DPD in the absence of iodide, N-bromo compounds do oxidize DPD in the absence of iodide.
  - d. Thus, carrying out the analysis in the absence of iodide gives the quantity of N-bromo compound. Repeating the analysis in the presence of iodide gives the total oxidant level, which is a measure of both the N-bromo and N-chloro compounds. The ratio of the N-bromo to total oxidant level represents the mole fraction of N-bromo compounds present in the final biocide solution.
  - e. One limitation of the above method is that since the test relies upon the slow reaction of N-Cl with DPD in the absence of iodide, the accuracy of the test when a large ratio of the N-halo compound is N-chloro (say 50% or more) can result in an erroneously high reading of N-bromo compound.
6. When the pH of the aqueous alkali metal sulfamate solution prior to bromine chloride addition has a pH above 13, and upon simultaneous addition of bromine chloride with sodium hydroxide in order to maintain the pH above 13, only about 80 mole % of the N-halo sulfamate compound formed was N-bromosulfamate. The remaining about 20 mole % N-halo compound formed was N-chlorosulfamate. Similar results were achieved regardless of whether the bromine chloride was pre-prepared or made inline.

7. The above results were consistent with the fact that bromine chloride exists as an equilibrium mixture of 20 mole % Br<sub>2</sub>, 20 mole % Cl<sub>2</sub>, and 60 mole % BrCl.
8. By contrast, keeping the other conditions the same, but instead of using BrCl, adding first bromine, then an equal amount of chlorine, only about 60 mole % of the N-halo sulfamate solution formed was N-bromosulfamate.
9. These differences in results demonstrate there is little or no oxidation of bromide to bromine by chlorine (reaction (1), below) or to hypobromite by hypochlorite (reaction (2), below) (either of which if occurred would prevent formation of N-chlorosulfamate), nor is there an N-chloro to N-bromo exchange occurring at pH above 13 (reaction (3), below). In other words, the following reactions do not occur at pH above 13:



Had any of these reactions occurred, then there would be no difference in using bromine or BrCl as the final percentage of N-bromosulfamate would have been about the same in both sets of experiments.

10. In summary, the overall reactions believed to occur upon bromine chloride addition to an alkali metal sulfamate solution at high pH are believed to be as follows:





11. The fact that reaction (2) (paragraph 9, above) does not occur at high pH is in contrast to what occurs at neutral pH. Specifically, the oxidation of bromide via the reaction:



is known to occur fairly rapidly in water at neutral pH. **EXHIBIT 2011**, p. 8 (cover counted as page 1), last paragraph. Accordingly, in water, bromine chloride appears to hydrolyze completely to hypobromous acid (HOBr). *Id.*

12. To my knowledge, the fact that oxidation of bromide does not occur at high pH, as demonstrated by the above experiments, was not known in the art prior to 2000.

Accordingly, I had no expectation prior to performing the above experiments that use of bromine chloride in the process would not be equivalent to using bromine. For the same reason, I also did not have any reason to expect that the process would be equivalent to using bromine.

13. Count 1 of Interference No. 105,230 is identical to claim 1 of Yang's U.S. Patent No. 6,287,473. Count 1 of Interference No. 105,222 is identical to claim 14 of Yang's U.S. Patent No. 6,423,267. I have been instructed by Moore's attorney's to interpret the scope of these counts in a manner which is consistent with the patent from which each count originates.

- a. As noted in my first declaration, one of skill in the art would interpret the "caustic solution" recited in claim 1 of the '473 patent and claim 14 of the '267 patent as having a pH higher than 14. **EXHIBIT 1001**, ¶¶ 44, 44(b), 93, and 93(b).

- b. Accordingly, one of skill in the art would interpret the “caustic solution” recited in Count 1 of the ‘222 interference and Count 1 of the ‘230 interference as having a pH higher than 14.
  - c. In view of the results of the experiments described above (¶¶ 4-10), I would expect that the immediate product of the process steps recited in Count 1 of the ‘230 interference, wherein sulfamic acid is used as the halogen stabilizer, to be a composition comprising N-bromosulfamate, N-chlorosulfamate, a bromide salt (for example, NaBr), and a chloride salt (NaCl). The composition could further comprise N,N-dibromosulfamate, N-bromo-N-chlorosulfamate, and N,N-dichlorosulfamate.
  - d. I would expect that the immediate product of the process steps recited in Count 1 of the ‘222 interference, wherein sulfamic acid is used as the halogen stabilizer, to be a composition comprising N-bromosulfamate and a bromide salt. The composition could further comprise N,N-dibromosulfamate.
14. Thus the immediate composition which results from the process recited in Count 1 of the ‘230 interference would contain the additional compounds N-chlorosulfamate and a chloride salt. The composition may further differ by containing N-bromo-N-chlorosulfamate, and N,N-dichlorosulfamate.
15. I have reviewed the Declarations of Dr. Shunong Yang, Dr. Jack Mills, and John A. Wojtowicz.
16. At ¶¶ 47-53 of the Yang Decl., Dr. Yang cites a number of references for the proposition that bromine and bromine chloride are interchangeable in the process recited in Count 1 of the ‘222 interference.

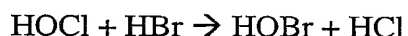
- a. The first reference, cited in ¶ 47, is Moore's U.S. Patent No. 6,068,861, col. 1, ll. 28-67. This section of the '861 patent describes the recognition in the art that both bromine and bromine chloride could be used as a biocide by direct introduction into a water system.
- b. The second reference, cited in ¶¶ 48-49, is a paper titled "The Chemistry of Bromine Chloride in Waste Water Disinfection." **EXHIBIT 2009**, p. 1. This reference only relates to the direct use of bromine chloride for wastewater treatment as a substitute for either bromine or chlorine. See, for example, **EXHIBIT 2009**, p. 6, "Conclusion".
- c. The third reference, cited in ¶ 50, is a product bulletin published by Dow Corporation. **EXHIBIT 2010**. This reference proposes bromine chloride as a viable alternative to chlorination for industrial cooling water systems. See, for example, **EXHIBIT 2010**, p. 1, col. 1, ¶ 1 and col. 2, ¶¶ 1-2 (not including bullet points).
- d. The fourth reference, cited in ¶ 51, is a brochure published by the Ethyl Corporation. **EXHIBIT 2011**. Similar to the above two references, the brochure advocates the advantages of using directly using bromine chloride over chlorine in water treatment. **EXHIBIT 2011**, p. 2 (counting cover as p. 1).

17. All of the references cited by Dr. Yang relate to use of bromine chloride in water treatment. Industrial water systems generally have a pH between 7 and 9. Thus the similarity in reactions which bromine and bromine chloride undergo in a water

system having a pH in this range cannot be predictably extrapolated to the high pH conditions called for in the “caustic solution” of Count 1 of the ‘222 interference.

18. At ¶ 47 of the Mills Decl., Dr. Mill cites his own paper (**EXHIBIT 2014**) for the proposition that in most bromination reactions, bromine chloride could be used instead of bromine. Thus, Dr. Mill asserts, it would have been obvious to use bromine chloride instead of bromine in the process recited in Count 1 of the ‘222 interference.

- a. Consistent with what was known in the art (see ¶ 11, above) Dr. Mills paper teaches that upon to addition to water (pH ~ 7), any bromide formed upon addition of BrCl to water would quickly be oxidized via the reaction:



**EXHIBIT 2014**, p. 164, col. 2, section on “Reactions of Bromine Chloride with Water”.

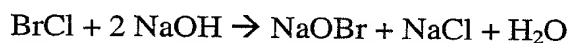
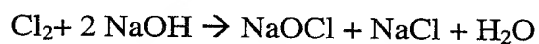
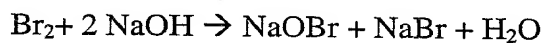
- b. I have reviewed **EXHIBIT 2014** and would agree with Dr. Mills that bromine chloride could be used as a substitute for bromine in the reactions discussed in this reference.
  - c. However, the process recited in Count 1 of the ‘222 interference is not discussed in Dr. Mills paper. Further, nothing in the paper suggests that bromine chloride and bromine are equivalent at the high pH called for in the “caustic solution” of Count 1 of the ‘222 interference.
19. As noted Dr. Mills paper, chlorination side reactions represented a significant concern since BrCl exists in an equilibrium mixture containing Br<sub>2</sub> and Cl<sub>2</sub>. **EXHIBIT 2014**, p. 160, col. 2, last paragraph. In my opinion, Dr. Mills paper would not have

suggested to one of skill in the art that N-chloro compounds would not be produced using bromine chloride under the high pH conditions called for in the “caustic solution” of Count 1 of the ‘222 interference or that the levels of N-bromo compounds would be equivalent to using bromine in the process.

20. At ¶¶ 49-54 of the Wojtowicz, Mr. Wojtowicz cites a number of references for the proposition that the substitution of bromine chloride in the process recited in Count 1 of the ‘222 interference would have been obvious.

- a. Based on my reading of these paragraphs, Mr. Wojtowicz fails to account for the fact that bromine chloride contains around 20% bromine, around 20% chlorine, and around 60% BrCl.
- b. Therefore, ¶ 50 of the Wojtowicz Decl., which asserts that both Br<sub>2</sub> and BrCl contain the same molar amount of “available bromine” (i.e., bromine in the +1 oxidation state) is factually incorrect. One mole of BrCl would only contain 0.8 moles of available bromine, whereas Br<sub>2</sub> would contain 1 mole of available bromine.
- c. Similarly, ¶¶ 51-52 of the Wojtowicz Decl., which compares the reactions of single Br<sub>2</sub> and BrCl molecules with base, fails to account for the fact that liquid bromine chloride comprises around 20% bromine and around 20% chlorine. Accordingly, ¶¶ 51-52 of the Wojtowicz Decl. are not predictive of the composition which results from substitution of bromine chloride for bromine under the high pH conditions called for in the “caustic solution” of Count 1 of the ‘222 interference.

- d. Since liquid bromine chloride contains around 20% bromine and around 20% chlorine, the following reactions would be expected to occur upon addition of bromine chloride to an aqueous base (NaOH) solution:



I hereby declare under penalty of perjury that all of the statements made by me herein are true.

Date: *Feb. 14, 2005*

380984v1

*B. Gary McKinnie*  
B. Gary McKinnie